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Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 18 DETERMINATION OF FREEZING POINT

(Second Revision)

ISO Title : Essential Oils — Determination of Freezing Point

Natural and Synthetic Perfumery Materials Sectional Committee, PCDC 18; Methods of Test Subcommittee, PCDC 18 : 1 [Ref : Doc : PCDC 18 (192)]

National Foreword

This Indian Standard (Part 18) (Second Revision), which is identical with ISO 1041-1973 'Essential oils — Determination of freezing point' issued by the International Organization for Standardization (ISO), was adopted by the Indian Standards Institution on the recommendation of the Natural and Synthetic Perfumery Materials Sectional Committee, and approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1952 entitled as 'Methods of test for essential oils' and subsequently revised in 1968 as 'Methods of sampling and test for natural and synthetic perfumery materials'. Taking cognizance of the need to incorporate instrumental methods of analysis now in vogue and to facilitate inclusion of additional test methods or changes in the existing test methods and also to align these test methods with the corresponding ISO standards, the committee concerned decided to revise and split the standard and publish individual test methods as separate parts of the original standard.

In order to facilitate proper reference of the method of test given in this revised part of IS : 326, Amendment No. 4 is being issued separately to the relevant clause of IS : 326-1968 'Methods of sampling and test for natural and synthetic perfumery materials (*first revision*) which would no longer be in force with the publication of this revision. The test method as given in this standard will supersede the test method given under **16.6** of IS : 326-1968.

In adopted standard certain terminology and conventions are not identical with those used in Indian Standards; attention is especially drawn to the following:

> Comma (,) has been used as a decimal marker while in Indian Standards the current practice is to use a point (.) as the decimal marker.
>
> Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'Indian Standard'.

Cross Reference

International Standard	*Corresponding Indian Standard*
ISO 212-1973 Essential oils — Sampling	**3.3** of IS : 326 (Part 1) 'Methods of sampling and test for natural and synthetic perfumery materials : Part 1 Sampling' (Technically Equivalent)
ISO 356-1977 Essential oils — Preparation of test sample	**3.5** of IS : 326 (Part 1) (Not equivalent)
ISO 653-1980 Long solid-stem thermometers for precision use	IS : 4825-1968 Laboratory and reference thermometers (Difference in details but technicaly equivalent)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the freezing points of essential oils. It is not applicable in the special case of essential oil of rose.

2 REFERENCES

ISO 212, *Essential oils — Sampling*

ISO/R 356, *Essential oils — Methods of test — Preparation of sample.*

3 DEFINITION

For the purpose of this International Standard, the following definition applies :

freezing point : Either the constant temperature, or the maximum temperature, observed when the oil, in a supercooled liquid state, liberates its latent heat of fusion.

4 PRINCIPLE

Slow and progressive cooling of the essential oil.

Observation of the variations in temperature as the oil passes from the liquid to the solid state.

5 APPARATUS

5.1 Calibrated thermometers, fulfilling the following requirements :

length of bulb : 10 to 20 mm

diameter of bulb : 5 to 6 mm

graduations : 0,1°C

It is essential that the set of thermometers used (see ISO/R 653) should permit the measurement of any temperature between — 20 and + 50°C.

The relevant International Standards for the essential oils indicate which thermometer shall be used.

5.2 Test tube, about 20 mm in diameter and 100 mm long.

5.3 Stout-walled test tube, about 30 mm in diameter and 125 mm long.

5.4 Apparatus assembly for determination of freezing point, consisting of a wide-mouthed container of about 500 ml capacity, provided with a bored cork or rubber stopper into which the stout-walled test tube (5.3) is inserted. The test tube (5.2) is fitted into the stout-walled test tube (5.3) by means of another bored cork or rubber stopper. Into the test tube (5.2) a thermometer (5.1) is inserted, so that the centre of its mercury bulb is located at the centre of the liquid.

A suitable apparatus assembly is shown in the figure.

6 SAMPLING

See ISO 212.

7 PROCEDURE

7.1 Preparation of test sample

See ISO/R 356.

7.2 Preliminary test

If necessary, first liquefy the essential oil by warming. Cool a few millilitres of the oil to be tested in a small test tube, and stir with a thermometer until solidification takes place.

Note the temperature and set aside in a cool place.

7.3 Determination

Fill the container (see 5.4) with water, melting ice or any suitable freezing mixture, so as to obtain a temperature 5°C lower than that noted in the preliminary test. Fit the stout-walled test tube (5.3) in its place.

Into the test tube (5.2), place 10 ml of the essential oil, liquefied if necessary, insert the thermometer and carefully cool the oil to the temperature indicated in the preliminary test. Now insert the test tube (5.2) in the stout-walled tube (5.3) of the apparatus and allow the temperature to fall a further 2°C.

Seed the oil with a trace of the solidified oil, obtained in the preliminary test, and stir vigorously with the thermometer, taking care to avoid the adhesion of particles to the walls of the tube. Observe the temperature variations as carefully as possible.

Note the observed temperature when the temperature curve in relation to time shows a maximum value, or a constant level for at least 1 min.

Remove the test tube (5.2) from the apparatus and again liquefy the essential oil. Repeat the determination until two consecutive results do not differ by more than 0,2°C. Average the two last readings to obtain the final figure.

8 EXPRESSION OF RESULTS

The freezing point is the maximum temperature observed at the end of the test. It is expressed in degrees Celsius to one decimal place.

9 TEST REPORT

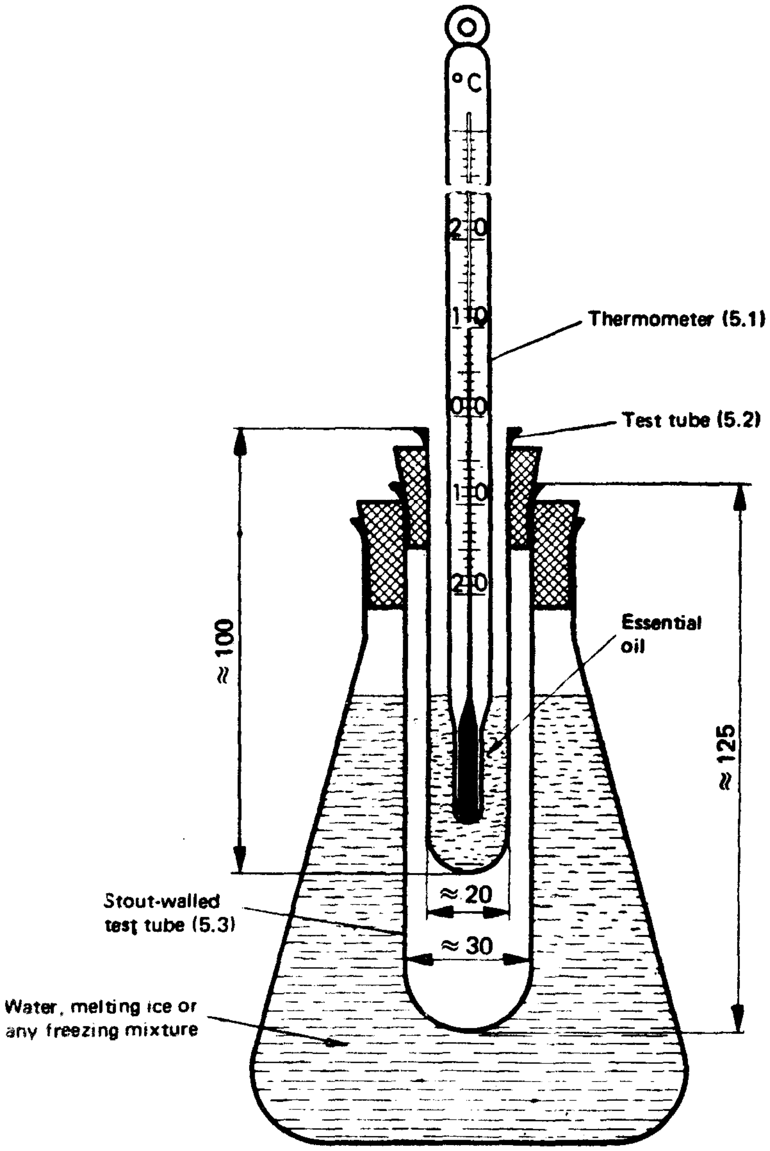
The test report shall include the following particulars:

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.



Dimensions in millimetres.

FIGURE — Suitable Apparatus Assembly for Determination of Freezing Point

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